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Investigation on Mechanical Properties of Nano Ferrous Composite

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Abstract

Nowadays metal matrix composites (MMCs) find application in various fields. Metals like Aluminium, Magnesium and Nickel etc., have been mixed with Carbon Nanotubes (CNTs) as reinforcement to prepare MMCs. There exists a growing interest in CNTs reinforced metal matrix composites (MMCs) due to the fact that CNTs can serve as very good reinforcing agents for MMCs. Reinforcement of the matrix metal with CNTs leads to enhancement in mechanical properties without any considerable increase in weight of the material. In this study, 3 different samples of multi-walled carbon nanotubes (MWNTs) reinforced iron composites were fabricated by using powder metallurgy technique by mixing the powdered iron and MWNTs manually and ball milling process, compacting the specimen using a cylindrical die and then sintering it. The compaction die was designed and fabricated using die steel. The various testing like tensile, compressive, hardness and chemical were carried out and surface characteristics were analysed. The test results show that the mechanical properties were found to be improved significantly when compared to unreinforced iron.

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1. Introduction

Carbon nanotubes (CNTs) are among those materials which are gaining more attention these days as reinforcements

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for composites due to their unique physical and mechanical properties combined with low density. CNTs are formed when one or many nano-sized sheets of graphene are rolled together. CNTs were first produced by Iijima in 1991 using arc-discharge evaporation method [1]. Nuno Silvestre et al [2] made a molecular dynamics study on the thickness and post critical strength of carbon nanotubes for various conditions and this study revealed that the post critical strength was almost zero for all types of carbon nanotubes for pure shortening whereas for pure twisting, all carbon nanotubes had negative post-critical strength, which became positive as the rotation was increased. Wan et al [3] revealed that the SWNT-matrix interphase is an important factor in determining the load transfer efficiency of the composite but this interphase affects the effective moduli and therefore the stiffness properties of the composite. They also found that the effective modulus of long SWNT fibers may be about twice as that for short SWNT fibers. Gibson et al [4] reviewed the vibrations of carbon nanotubes and their composites and have arrived at a conclusion that the unique properties of vibrating CNTs are being applied to develop new nanomechanical devices and instruments like sensors, actuators, high frequency resonators and oscillators, field emission devices etc. Vibrations of CNTs induced by ultrasonics, microwave and some other high propagation wave are being used to improve dispersion of CNTs in matrix phase of resin-matrix composites but some more work has to be carried out to make this process optimized. Savvas et al [5] demonstrated that there was reduction in loss factor due to CNT waviness which led to the conclusion that to improve damping properties of the CNT reinforced composites, straightening of CNTs is important.

Ajayan and Zhou [6] reviewed that the remarkable physical properties of CNT structures make them a very unique material with a whole range of promising applications. Tadayuki Tsutsui [7] studied the iron-based sintered parts and concluded that sinter hardened materials produced without quenching process are in practical use. Prabhu and Vinayagam [8] found that a nano material, especially MWNTs is used in the machining process like grinding to improve the surface characteristics from micro to nano level. Yufeng Wu and Gap-Yong Kim [9] evaluated the Al6061-CNT composite by mechanical alloying and obtained a maximum value of hardness of 87.5 HV at 620˚C. Lai-Xue Pang et al [10] fabricated a dense Fe_3Al-CNT composite by Spark Plasma Sintering (SPS) method at a pressure and temperature of 30 MPa and 1273 K respectively. For 5 vol. % of CNT, the maximum value of microhardness and compressive yield strength of 8.7 GPa and 3175 MPa respectively was achieved. Bradbury et al [11] reported that the maximum possible hardness of MWNTs reinforced aluminium composites synthesized by milling and hot pressing was found to be 151 HV obtained at 6 wt. % of CNT. Dehong Lu et al [12]found that 0.1% $A1_2O_3$ and 0.2% CNTs reinforced AZ31 magnesium alloy composite showed lower wear for load more than 1.95 MPa and 0.2% Al_2O_3 and 0.1% CNTs reinforced AZ31 composite had an improved maximum hardness value than that of AZ31 up to 1.4 times.Kim et al [13] compared the friction and wear characteristics of Al-CNT composite manufactured by Hot Pressing (HP) and Spark Plasma Sintering (SPS) methods and concluded that the composite fabricated by SPS method had better properties than the one fabricated by HP method. Choi et al [14] observed that the yield strength and % plastic elongation to failure improved in aluminium nanocomposite when it was reinforced with 5 wt. % Si alloy and 3 vol. % MWNTs. According to Liu et al [15], the CNTs showed improved strength when ball milling time was upto 6 hours and the strength deteriorated with further increase in ball milling time. Kwon et al [16] observed that the Vicker's hardness and bending strength of dual nanoparticulate (n-SiC and CNT) reinforced aluminium composite enhanced when milling time was increased and 5 times the hardness of pure Al 6061 alloy was obtained in the composite. Sebastian et al [17] concluded that there was no improvement in properties of MWNT-Ni bulk composite beyond 3 wt. % of CNT as CNTs reagglomerated due to large volume fraction. Pe´rez-Bustamante et al [18] showed that the Al 2024-CNT composites showed improved wear properties when the CNT content was as high as the order of 5 wt. %. Esawiand Borady [19] studied properties of CNT-Al strips and the Al–0.5 wt. % CNT strips exhibited improved tensile strength, yield strength, Young's modulus as well as lower density. In this study, iron powders reinforced with MWNTs were fabricated by powder metallurgy technique and various mechanical and chemical tests were conducted. For preparation of specimen for various tests, a die set was designed and fabricated using die steel. Review on CNT Reinforced Aluminium and Magnesium Matrix Composites has been done and suggesting new cost effective production method must be developed for manufacturing CNT.[22].

Nomenclature

CNTs	Carbon Nanotubes
MMCs	Metal Matrix Composites
SWNTs	Single-Walled Carbon Nanotubes
MWNTs	Multi-Walled Carbon Nanotubes
CVD	Chemical Vapour Deposition
HV	Vicker's Hardness
BHN	Brinell hardness number
HRC	Rockwell Hardness 'C' Scale
HP	Hot Pressing
SPS	Spark Plasma Sintering
TEM	Transmission Electron Microscopy
PCA	Process Control Agent
UTM	Universal Testing Machine
A_i	Area of Indentation
D	Indentor Diameter
d	Indentation Diameter
P	Load Applied
HB	Brinell hardness

2. Experimental Setup:

2.1. Materials used:

The materials used in this study were iron powders and MWNTs. The iron powders used were 99.50% pure, irregular powders with melting point of 1535˚C and boiling point of 3000˚C. CNTs may be of two types namely Single walled Carbon nanotubes (SWNTs), when a single sheet of graphene is rolled and multi-walled Carbon nanotubes (MWNTs), when multiple graphene sheets are rolled. CNTs are generally produced by Arc-Discharge method, Laser Ablation method or Chemical Vapour Deposition (CVD) method. Nanotubes can have diameters ranging from 1 to 100 nm, lengths of up to few millimetres, and densities as low as ~1.3 g/cm^3 with a Young's modulus value of about 1TPa [20]. The wrought density, apparent density and particle size used in this study were 7.86 g/cm^3, 3.6 g/cm^3 and 250 mesh respectively. The properties of MWNTs used include purity > 95% on trace metal basis, melting point of 3652-3697˚C, density of ~2 g/cm^3 and dimensions of 10-30 nm of outer diameter, 2-6 nm of inner diameter and 15-30 μm of length. The impurities present in the MWNTs used were amorphous carbon of about < 3%, which was discovered by using TEM analysis.

2.2. Specimen Preparation:

In the present work, powder metallurgy was the technique used to fabricate the various test specimens. Powder metallurgy technique generally involves 3 basic steps: Blending, compaction and sintering. Blending is done in order to mix the powder particles to mix well with each other and to obtain a desired powder size. Compaction is done to get the near net shape of the specimen. In compaction stage, the powders were compacted in a die of desired shape along with a suitable binder material. The sintering process is done in order to transform the compacted mechanical bonds to strong metallic bonds.

The iron powders and MWNTs were blended together manually for 3 hours and then by ball-milling for 30 min. Manual process was done for 100g of powders by using pestle and mortar. The ball-milling process was carried out

using 250ml stainless steel mixing jars and stainless steel milling balls of 5mm diameter. The ball-to-powder weight ratio was maintained at 10:1 for ball-milling and process was carried out at a speed of 400 rpm. To minimize cold welding between iron powders and also to prevent adhesion between powders, steel balls and walls of the jar, isopropyl alcohol was used as process control agent (PCA). 1 wt % of liquid binder was added in order to improve flow characteristics and compressibility of the powder mixture and paraffin wax was added to improve green strength of the component. The powder mixture was then compacted using a die of 20 mm diameter (Fig. 1) at a compacting pressure of 711.80 MPa at room temperature in a universal testing machine (UTM). 3 different samples were produced and tested in this study. The % CNT of the 3 samples is shown in Table 1. The green components prepared were then sintered by pre-heating them to a temperature of ~0.8 times their melting point. The initial temperature for sintering was set as 300˚C and 100˚C rise in temperature was made for time interval of 1 hour up to 800˚C and then gradually annealed to atmospheric temperature. The compacted green components and one of the sintered components are shown in Fig. 2 (a) and (b).

Table 1. Percentage of CNT in various samples.

Specimen	% CNT
Nano Iron Sample 1	Iron + 0.25% CNT
Nano Iron Sample 2	Iron + 0.5% CNT
Nano Iron Sample 3	Iron + 0.67% CNT

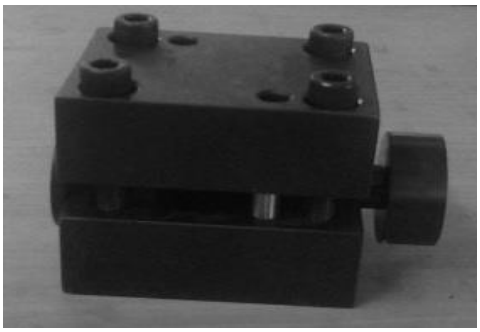

Fig. 1. Die used for compacting of powders.

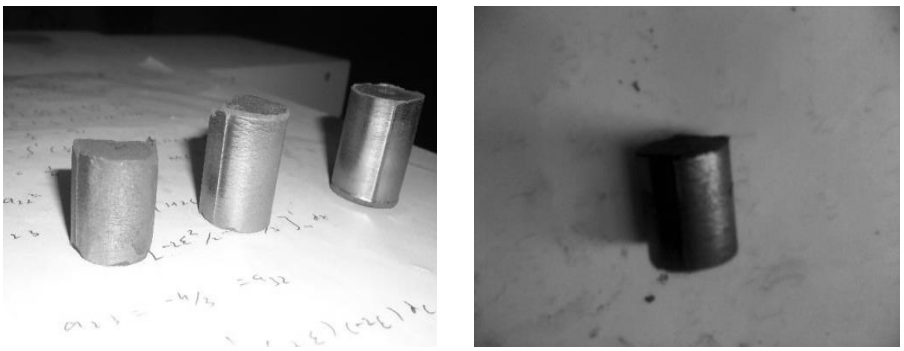

Fig. 2. (a) Compacted green components, (b) Sintered Nano iron specimen.

3. Experiments, Results and Discussion:

3.1. Hardness Test:

Hardness of a material is the ability of that material to withstand any indentation or abrasion. It can generally be determined by using Rockwell, Brinell or Vicker's hardness testing machine. In this method, a small indenter was pressed against the material by applying suitable load and hardness of the material was directly related to the diameter of indentation. The hardness test for samples with and without CNT was conducted in both Rockwell and Brinell hardness testing apparatus. For Brinell hardness, a load of 3000 kg was applied using a 10mm diameter spherical indentor and indentation diameter of 6mm for iron sample and diameters of 5.4mm, 4.6mm and 4mm for samples 1, 2 and 3 were obtained respectively. This hardness was determined by using the formula as stated below.

$$A_i = \frac{\pi D}{2}[D - \sqrt{D^2 - d^2}] \ \text{mm}^2 \quad (1)$$

$$HB = \frac{P}{A_i}\text{BHN} \quad (2)$$

The Brinell hardness for Iron sample was found to be 95.49 BHN and nano iron samples had Brinell hardness of 120.62, 170.399 and 228.76 BHN respectively, which shows the effect of CNT on the hardness of the samples.
For Rockwell hardness, for a load of 150 kgf on both the specimen, the value on Rockwell C scale for iron sample was found to be 60 HRC and for nano iron samples were found to be 63, 68 and 70 HRC respectively. This proves the improved hardness that was obtained from Brinell scale and Rockwell 'C' scale was found to be due to the reinforcement of CNT with the matrix. The values of Brinell and Rockwell hardness are given in Table 2.

Table 2. Brinell and Rockwell hardness values of Iron and Nano Iron samples.

Specimen	Brinell Hardness (*BHN*)	Rockwell Hardness (*HRC*)
Iron	95.49	60
Nano Iron Sample 1	120.62	63
Nano Iron Sample 2	170.399	68
Nano Iron Sample 3	228.76	70

3.2. Tensile Test:

Tensile strength of a material is defined as the maximum stress the material can withstand without failure or rupture. In this study, 3 specimens of nano iron were prepared and tensile load was applied on each specimen separately using a universal testing machine (UTM). The values of tensile strength of different samples are shown in Table 3. It was observed that a maximum tensile strength of 1.051GPa was obtained in sample 3. This shows that the tensile strength of the iron specimen increases with increase in % CNT content in the composite.

Table 3. Tensile strengths of various samples.

Specimen	Failure Load (*kN*)	Tensile Strength (*MPa*)
Iron	281.986	897.59
Sample 1	300.439	956.33
Sample 2	310.788	1002.82
Sample 3	315.045	1051.27

3.3. Compression Test:

Compressive strength of a material is defined as the ability of material to withstand compressive loading without failure. Compression test is done by compressing a standard specimen using a UTM. In this study, 3 nano iron samples were prepared along with one iron sample. On testing the samples for compressive strength, the sample reinforced with CNT showed some significant improvement when compared with unreinforced sample. The iron sample had a compressive strength of 751.21 MPa where as the CNT reinforced iron samples had improved compressive strengths of 792.59 MPa, 853.07 MPa and 916.73 MPa. A similar result was obtained by J.Y. Suh and D.H. Bae [21], with 4 vol. % MWNT reinforced Fe powders when compared to unreinforced Fe, both produced by hot pressing. They showed that increase in vol. % of MWNT increased the compressive strength of the composite. The relationship between applied load and stroke length for various samples is shown in Fig. 3 (a) and (b).

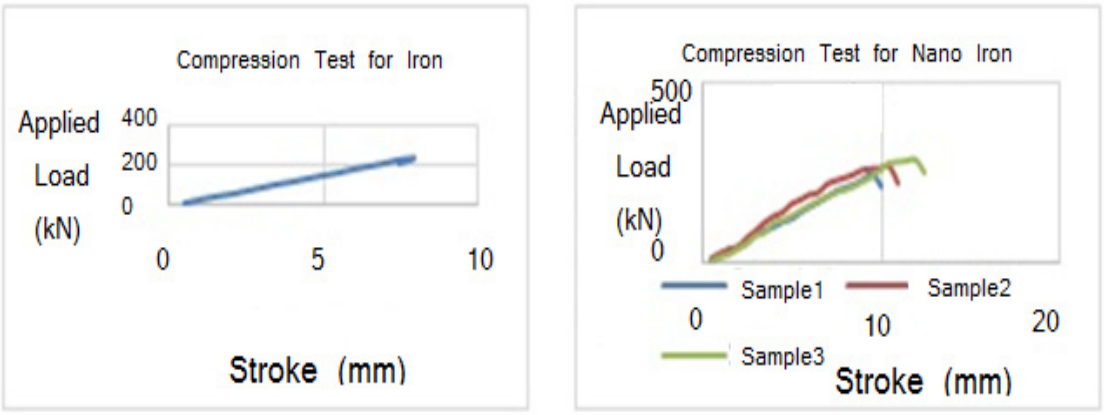

Fig. 3. (a) Compression Test graph for iron, (b) Compression test graph for nano iron specimens

The values of compression test for various samples are shown in Table 4.

Table 4. Compression test results for Iron and Nano Iron.

Specimen	Compressive Strength (MPa)
Iron	751.21
Sample 1	792.59
Sample 2	853.07
Sample 3	916.73

3.4. Micro measurement test:

Micro measurement test was conducted on the samples using Material Plus software to compare the density and porosity of the nano iron with the values of iron. According to the test results, for iron, the % of white region was 37.39% (Fig. 4 (a)) whereas that of nano iron was 39.67% (Fig. 4 (b)). This proves the fact that the properties of the material increases without any significant improvement in density of the material when reinforced with CNTs. The porosity of iron was 86.6% (Fig. 5 (a)) with maximum and minimum perimeter as 31637.4306 microns and 10.5263 microns respectively with maximum and minimum area of 10.5263 sq. microns and 3698116.3435 sq. microns respectively. Compared to this, the porosity of nano iron was observed to be 77.31% (Fig. 5 (b)) with 74559.0769 microns and 10.5263 microns of maximum and minimum perimeter and 10.5263 sq. microns and 3472382.2715 sq. microns of maximum and minimum area respectively.

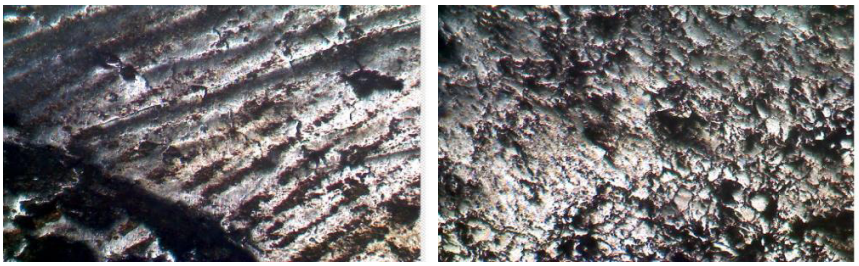

Fig. 4. (a) Microstructure for density of Iron, (b) Microstructure for density of Nano Iron.

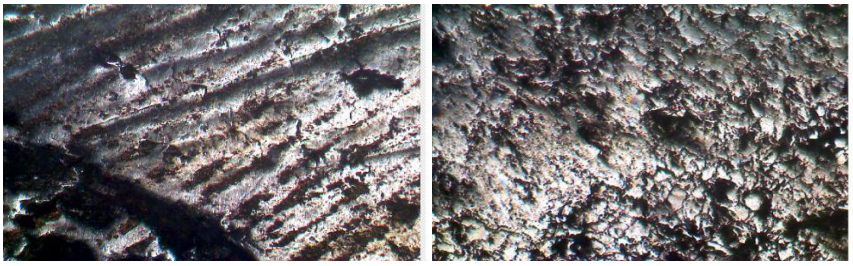

Fig. 5. (a) Microstructure for porosity of iron, (b) Microstructure for porosity of Nano iron.

3.5. Finite Element Analysis:

The stress and thermal analysis of the unreinforced iron and CNT reinforced iron was analysed using ANSYS software. During stress analysis, one end of the rod was constrained in all directions and compressive force of 100 kN was applied on the other end. The iron specimen had a permissible load of 6.21 N and ultimate strength of 6.986 N/mm^2 as shown in Fig. 6 (a). Once after the permissible load value was reached, the nano iron rod was subjected to crack formation and a maximum deformation of 3.289 mm at the point of load application and a minimum deformation at the other end was observed, which is shown in Fig. 6 (b). The maximum stress induced in the CNT reinforced iron was 21051 N/mm² which was lower and safe when compared with the value of unreinforced iron of 26022 N/mm².

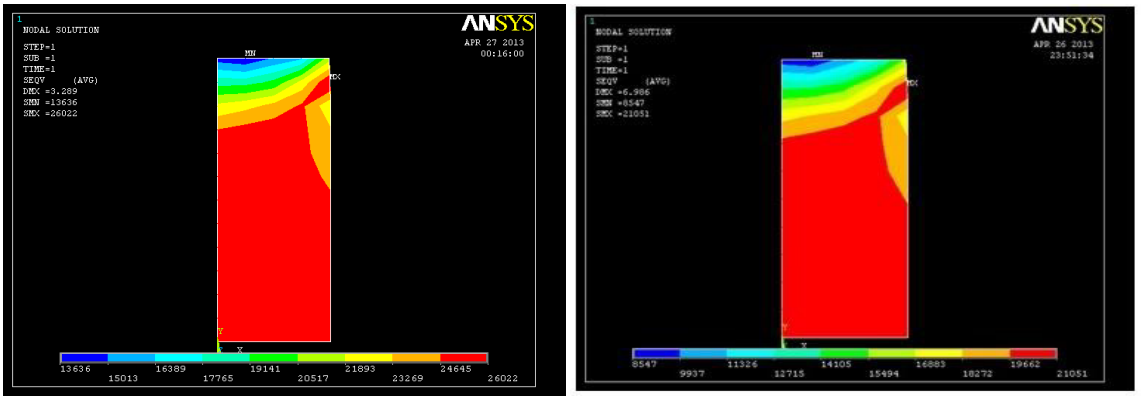

Fig. 6. (a) Stress Analysis of Iron using ANSYS, (b) Stress Analysis of Nano Iron using ANSYS.

The thermal analysis in ANSYS (Fig. 7 (a) and (b)) showed that the Nano iron was under safe when compared to unreinforced iron. In this case, the iron rod was subjected to all types of thermal loads and it was found that the unreinforced component had thermal stability under the same temperature with a value of 71.11^0C whereas the Nano iron had a value of 53.33^0C. Hence it was found that the temperature had been enormously stable under the stated conditions (25%).

Fig. 7. (a) Thermal analysis of iron using ANSYS, (b) Thermal analysis of nano iron using ANSYS.

4. **Conclusion:**

In this study, iron powders reinforced with 3 different % content of MWNTs were successfully fabricated by using powder metallurgy technique. The samples produced were subjected to various mechanical tests and the results were compared with the unreinforced iron powders fabricated. The various results are as follows:

1. The Brinell hardness for iron, samples 1, 2 and 3 were found to be 95.49, 120.62, 170.399 and 228.76 BHN respectively.
2. The Rockwell hardness for iron, sample 1, sample 2 and sample 3 were found to be 60, 63, 68 and 70 HRC respectively.
3. The maximum value of tensile strength was obtained for sample 3 and the value is 1051.27 MPa.
4. The compressive strength of iron improved from 751.21 MPa to a maximum 916.73 MPa for sample 3.
5. No significant improvement in density was observed in nano iron.
6. The nano iron had a low and safe stress value of 21051 N/mm² when compared to that of iron (26022 N/mm²).
7. Iron was thermally stable up to a temperature of 71.11^0C whereas nano iron was thermally stable only up to a temperature of 53.33^0C.

These results prove that the CNTs can be used as a good reinforcing agent for metal matrix composites used for various automotive and structural applications.

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